AD 45694

UNCLASSIFIED

45 644

WADC TECHNICAL REPORT 54-62

BUTYL INNER TUBE COMPOUND FOR AIRCRAFT TIRES

EMMETT B. REINBOLD

THE GENERAL TIRE & RUBBER COMPANY

JULY 1954

WRIGHT AIR DEVELOPMENT CENTER

WADC TECHNICAL REPORT 54-62

BUTYL INNER TUBE COMPOUND FOR AIRCRAFT TIRES

Emmett B. Reinbold

The General Tire & Rubber Company

July 1954

Materials Laboratory
Contract No. AF 33(600)-22796
RDO No. 617-12 (C-K)

Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

Carpenter Litho & Prtg. Co., Springfield, O.
200 - 12 October 1954

FOREWORD

This report was prepared by The General Tire & Rubber Company, under USAF Contract No. AF 33(600)-22796. The contract was initiated under Research and Development Order No. 617-12(C-K), "Compounding of Elastomers," and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Major H. C. Hamlin and Lt Kelble acting as project engineers.

ABSTRACT

This report deals with the development of a butyl rubber compound with requisite physical properties for fabrication into inner tubes for aircraft tires.

A large number of plasticizers of widely divergent chemical characteristics were evaluated, with special emphasis placed on their low temperature properties in butyl rubber and including the technique of using high black, high plasticizer with a high viscosity elastomer. The effect of zinc oxide content in the formula was determined. Carbon blacks of all commercially available types were compared and the effect of the various blacks on physical properties, including low temperature characteristics were determined. On blacks producing highest tensile values, series of tests were made to determine the loading which produces the maximum physical properties. A comparison of low temperature properties is made of the available commercial butyl rubbers. An extensive investigation of an outstanding low temperature material, Silicone, was carried out.

On plasticizers which produced adequate low temperature flexibility in butyl rubber a study is made on volatility and migration of the plasticizer from cured compound and then low temperature properties determined following migration treatment of cured tensile sheets. In all cases, plasticizers which produced the target low temperature requirements showed poor low temperature properties following migration treatment.

The effect of inorganic acceleration and of the recently recommended processing technique of high temperature mixing was investigated.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. Whitmore

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

TABLE OF CONTENTS

	Page
INTRODUCTION	1
DISCUSSION	2
SUMMARY AND CONCLUSIONS	8
TESTING METHODS AND PROCEDURES	9

LIST OF TABLES

Table		
	A. PLASTICIZERS INCOMPATIBLE WITH BUTYL	
I.	Modulus @ 300% - Pounds Per Square Inch	12
II.	Tensile @ Break - Pounds Per Square Inch	12
III.	Elongation @ Break - Per Cent	13
IV.	Tear - Pounds Per Inch	13
V.	Hardness (Shore Durometer Type A)	14
VI.	Rebound - Embrittlement - TR	15
VII.	TR - Temperature Retraction	16
	B. SILICONE DC 510 FLUID AND ESTER PLASTICIZER IN BUTYL	
VIII.	Stress - Strain, Tear And Shore Durometer	17
IX.	Rebound - Embrittlement - TR	18
X.	TR - Temperature Retraction	19
	C. PLASTICIZERS WITH DEFICIENT FREEZE RESISTANCE	
XI.	Modulus @ 300% - Pounds Per Square Inch	20
XII.	Tensile @ Break - Pounds Per Square Inch	21
XIII.	Elongation @ Break - Per Cent	21
XIV.	Tear - Pounds Per Inch	22
XV.	Hardness (Shore Durometer Type A)	22
XVI.	Rebound - Embrittlement - TR	23
XVII.	TR - Temperature Retraction	24
XVIII.	TR - Temperature Retraction For Forum 40 Plasticized Stocks Following Migration Tests.	25
	D. PLASTICIZERS WITH SATISFACTORY FREEZE RESISTANCE	
XIX.	Modulus @ 300% - Pounds Per Square Inch	26
XX.	Tensile @ Break - Pounds Per Square Inch	27
XXI.	Elongation @ Break - Per Cent	27
XXII.	Tear - Pounds Per Inch	28
XXIII.	Hardness (Shore Durometer Type A)	28
XXIV.	Rebound - Embrittlement - TR	29
XXV.	TR - Temperature Retraction	30
XXVI.	TR - Temperature Retraction After 4 Weeks Migration at 158°F.	31

LIST OF TABLES (Cont'd)

Table		Page
	E. HIGH PLASTICIZER HIGH CARBON BLACK	
XXVII.	Modulus @ 300% - Pounds Per Square Inch	33
XXVII.	Tensile @ Break - Pounds Per Square Inch	34
XXVIX.	Elongation @ Break - Per Cent	34
XXX.	Tear - Pounds Per Inch	35
XXXI.	Hardness (Shore Durometer Type A)	35
XXXII.	Rebound - Embrittlement - TR	36
XXXIII.	TR - Temperature Retraction	37
	F. SPECIAL ACTIVATING AGENTS - POLYAC AND TELLURAC	
XXXIV.	Stress, Strain, Tear, Shore Durometer, Rebound, Embrittlement, TR	39
XXXV.	TR - Temperature Retraction	39
	G. COMMERCIAL CARBON BLACKS IN BUTYL	
XXXVI.	Stress, Strain, Tear, Shore Durometer	40
XXXVII.	Rebound - Embrittlement - TR	41
XXXVIII.	TR - Temperature Retraction	42
	H. VARIABLE CARBON BLACK LOADING IN BUTYL	
XXXIX.	Stress - Strain	43
XL.	Tear - Shore Durometer	44
XLI.	Rebound - Embrittlement - TR	45
XLII.	TR - Temperature Retraction	45
	I. VARIABLE ZINC OXIDE IN BUTYL	
XLIII.	Stress - Strain, Tear, Shore Durometer	46
XLIV.	Rebound - Embrittlement - TR	47
XLV.	TR - Temperature Retraction	47
	J. VARIABLE ISOPRENE CONTENT IN BUTYL	
XLVI.	Stress - Strain, Tear, Shore Durometer	48
XLVII.	Rebound, Embrittlement, TR - and Comparison TR and Embrittlement with State of Cure	49
XLVIII.	TR - Temperature Retraction	50
	K. PEROXIDE CURATIVES IN BUTYL	
XLIX.	Stress - Strain, Tear, Shore Durometer	51
L.	Rebound - Embrittlement - TR	52
LI.	Mooney Scorch (MS @ 250°F.)	52
LII.	TR - Temperature Retraction	53

Table		Page
	L. HIGH TEMPERATURE (400°F.) PROCESSING	
LIII.	Stress - Strain, Tear, Shore Durometer and Rebound. Rebound at Room Temperature and at 212°F.	55
LIV.	Stress - Strain and Tear @ 212°F.	56
LV.	Rebound, Embrittlement, TR	57
LVI.	TR - Temperature Retraction - Normal and After 4 Weeks Migration at 158°F.	57
	M. VOLATILITY OF PLASTICIZERS	
LVII.	Volatility of Plasticizer Itself and Volatility of Plasticizer in Vulcanized Butyl Inner Tube Compound	58
	N. MIGRATION OF PLASTICIZERS	
LVIII.	Per Cent Plasticizer Migrated from Cured Butyl Inner Tube Compound at Intervals 1 Day to 28 Days	59
	O. MIGRATION, EFFECT OF ON LOW TEMPERATURE PROPERTIES	
LIX.	Low Temperature Properties, Both Before and After Migration Tests on Butyl Tube Compounds With and Without Plasticizer	60

INTRODUCTION

Natural rubber, the elastomer currently used in inner tubes for aircraft tires is transported to this country by maritime shipping from great distances and in the event of international crisis, there is a possibility this supply could be cut off or interrupted. Under such circumstances it is highly desirable to have available a material which will be satisfactory for inner tubes for aircraft tires, and since at this writing butyl rubber is the elastomer with best air retentivity, it is the logical material for this application. One of the deficiencies of butyl rubber in inner tubes is lack of resilience at low temperatures.

The objective of this contract is the development of a butyl rubber compound which is flexible at temperatures down to -70°F. In compound development for low temperatures, the usual approach is the selection of suitable plasticizers. A satisfactory plasticizer must not only produce satisfactory initial low temperature properties but must not migrate into other stocks into which it will come in contact and must not be lost through extreme volatility.

Only through the use of the finer particle carbon blacks can the requisite physical properties be met.

As to processing of butyl rubber, this is a relatively new art and it is reasonable to expect that more will be learned which will help overcome some of the deficiencies of this elastomer. By virtue of General Tire's position in the rubber industry as a fabricator of elastomers commercially available and not a producer or manufacturer engaged in the development of new butyl polymers, this development is limited to a study of the butyl rubber which is commercially available. Obviously the method of attack is limited to compound development. While development of a new elastomer is a possible solution to the problem, it is beyond the scope or facilities of this contract.

DISCUSSION

On account of certain physical requirements deemed essential in service conditions to which they are subjected, inner tubes for aircraft tires are now fabricated from natural rubber. Despite superior air retention of butyl inner tubes, butyl compounds currently used in civilian applications are inadequate for military service, due largely to deficiencies in physical properties and low temperature flexibility. On account of the extreme importance of having an elastomer which is domestically available and which does not require importation, especially under adverse international situations, it is highly desirable that a butyl inner tube compound be developed which will possess the physical properties and low temperature flexibility requirements specified for inner tubes for aircraft tires. The retentivity of air with butyl rubber is so outstanding as compared to other elastomers from which inner tubes might or could be fabricated that butyl was selected as the subject for this investigation. Of the deficiencies above mentioned for butyl compounds, it was agreed at the start of this investigation that the most important requirement of this project was solution of the low temperature flexibility problem. It was furthermore agreed that certain compromises could be made on the other physical specifications provided a compound was developed which possessed the required low temperature flexibility.

In the early period of butyl tube manufacture one of the major deficiencies of inner tubes formulated from this elastomer was low temperature buckling, particularly in passenger tires where the tire and tube underwent considerable deflection. This condition was not considered a serious problem in truck tire inner tubes. Through an extensive development program conducted by some of the inner tube manufacturers and producers of butyl rubber, low temperature buckling has been almost or entirely eliminated. This development was accomplished by the addition of relatively large quantities of moderately low viscosity mineral oil, and in order to prevent stocks in process from becoming soft and mushy, higher viscosity butyl polymers were made available to the inner tube manufacturers for use with the high oil compounding technique. The introduction of high oil/high viscosity polymer in inner tube compounds was accompanied by increased carbon black loadings, which also assisted in maintaining the desired stiffness in the uncured state. Through this development it was noted that numerous plasticizers which are capable of imparting desirable low temperature properties to a butyl inner tube compound have a tendency to migrate from the butyl inner tube into adjacent stocks. It was required, therefore, in this development that any plasticizer in order to be of value in this application must not migrate into other stocks with which the inner tube comes in contact. And for purposes of migration evaluation, the experimental compounds were placed in contact with a natural rubber airplane tire carcass compound.

In the development of elastomeric compounds for low temperature service, the result is usually achieved by selection of a proper plasticizer, and for this reason the major portion of this development deals with plasticizer evaluation. Most of the materials used in this development, therefore, were plasticizers which had demonstrated outstanding low temperature characteristics in other elastomers or had promise because of desirable viscosities and pour points at low temperature. An essential requirement for materials used in butyl formulations is that they possess little or no chemical unsaturation. However, due to insufficient information furnished by some suppliers regarding characteristics or constitution of

their materials, some items were investigated which were incompatible and on account of chemical unsaturation failed to cure.

Based on observations and data obtained, the plasticizers investigated in this development were classified in the following three categories:

a. Plasticizers incompatible with butyl rubber.

b. Plasticizers with inadequate freeze resistance.

c. Plasticizers producing adequate freeze resistance.

PLASTICIZERS INCOMPATIBLE WITH BUTYL RUBBER

Materials in this category are plasticizers described by the suppliers as glycol derivatives, triglycol, polyalkylene glycol, glycol esters, polyesters, polyethers, ricinoleates and derivatives, tributoxyethyl phosphate, tributyl phosphate, dibenzyl ether, dibutyl aconitate and Atlas Powder's Pycal 70.

1. #4141 - C. P. Hall Co.

   A glycol fatty acid ester. Supplier states this material is similar to plasticizer SC.

2. Plasticizer SC - Harwick Standard Chemical

   A triglycol ester of a vegetable oil fatty acid.

3. Plasticizer DP-520 - Harwick Standard Chemicl

   A polyester.

4. KP-140 - Ohio Apex Inc.

   Tributoxyethyl phosphate.

5. P-1 - Baker Castor Oil Co.

   Methyl ricinoleate.

6. P-8 - Baker Castor Oil Co.

   Glyceryl triaceto ricinoleate.

7. TP 90 B - Thiokol Corp.

   High molecular weight polyether.

8. Ucon LB-65 - Carbide & Carbon Chemicals

Polyalkylene glycol. Water insoluble, low molecular weight and low pour point.

9. Dibutyl maleate - Carbide & Carbon Chemicals

10. Tributyl phosphate - Ohio Apex Inc.

11. Dibenzyl ether - Heyden Chemical Co.

12. Pycal 70 - Atlas Powder Co.

No information or description disclosed.

13. Dibutyl aconitate - C. P. Hall Co.

PLASTICIZERS WITH INADEQUATE FREEZE RESISTANCE

1. Plasticizer MT-511 - Harwick Standard Chemical

Condensation product of a polyhydric alcohol with an alpha - omega dicarboxylic acid.

2. VR-1 Ester (Genplast) - General Tire & Rubber Co., Chemical Division

A sebacic acid ester.

3. Dibutyl sebacate - Resinous Products Corp.

4. Adipol ODY - Ohio Apex Inc.

n-Octyl decyl adipate.

5. Ohopex R-9 - Ohio Apex Inc.

Not disclosed.

6. KP-555 - Ohio Apex Inc.

Bisdimethylbenzyl ether.

7. Monoplex S-71 - Rohm & Haas

Monomeric ester.

8. Silicone oils.

These oils are linear polymers with alternate atoms of silicon and oxygen. Organic groups are attached to the silicon atoms.

The silicones are characterized by retention of fluidity at very low temperatures.

a. Silicone L-41 is the commercial designation of the silicone oils where the organic groups are ethyl.

b. Silicone L-45 is the commercial designation of the silicone oils where the organic groups are methyl.

c. Silicone DC-510 fluid. This is a methyl ethyl derivative. Freezing point ranges below -70°C. and is recommended where extremely low temperatures are involved. This silicone exhibits the best low temperature properties of all the silicone fluids. Viscosity is 50 centistokes.

PLASTICIZERS PRODUCING ADEQUATE FREEZE RESISTANCE

The requirements on low temperature specified in MIL-T-5014B for inner tubes for aircraft tires specify flexibility at -70°F. (-57°C.) as measured by ASTM Test D746-44T. This requirement was met by the following plasticizers:

1. Plasticizer 3890-A - C. P. Hall Co.

   Dicarboxylic acid ester.

2. Adipol 2 EH - Ohio Apex Inc.

   Di - 2 - ethyl hexyl adipate.

3. 10-A plasticizer - Ohio Apex Inc.

   Di-iso-octyl adipate.

4. Di-2-ethyl hexyl ether - Carbon & Carbide Chemical Co.

5. Butyl Cellosolve pelargonate - C. P. Hall Co.

   Ester of n-nonylic acid.

6. Butyl Carbitol pelargonate - C. P. Hall Co.

7. Diisobutyl azelate - C. P. Hall Co.

   Ester of nonanedioic acid.

8. Monoplex DOS - Rohm & Haas

   Dioctyl sebacate.

9. Hexyl ether - Carbide & Carbon Chemical Co.

10. Trioctyl phosphate - Carbide & Carbon Chemical Co.

Brittle points for all of the above plasticizers are almost alike, that is, there is but a few degrees difference between the poorest and the best. The same also holds true for the physical properties in that no major differences are noted for any of the above materials. It is interesting to note, however, that almost without exception the ester plasticizers produce a more resilient compound than that produced by a paraffinic type mineral oil used in manufacture of inner tubes for truck and passenger tires.

Additional improvement can be obtained in low temperature flexibility with some plasticizers by the use of increased plasticizer. With increased plasticizer the additional increment is accompanied by a corresponding increase in carbon black in order to maintain stock plasticity which will permit normal factory processing. Normal plasticizer is considered as in the range of 20 to 25 parts per 100 parts rubber hydrocarbon.

There was some overlapping between groups 2 and 3 in that with lower loadings (20 parts) the brittle point of the resultant compound was unsatisfactory; whereas, with increased amount of plasticizer a satisfactory brittle point was obtained. Therefore, in the data tables some plasticizers may appear in both groups 2 and 3, depending on concentration of plasticizer used. For instance, C. P. Hall 3890-A produces satisfactory brittle point with 25 parts plasticizer but is deficient with 20 parts plasticizer.

Materials producing satisfactory low temperature properties were further evaluated for:

1. VOLATILITY of the plasticizer from the cured compound.

2. MIGRATION of the plasticizer from cured butyl stock to an adjacent natural rubber stock of the type airplane inner tubes would contact. For the adjacent natural rubber stock, compound K-8, General Tire's airplane tire carcass compound was used, with testing details as described under heading "testing methods and procedures."

Tensile slabs placed in contact with natural rubber carcass stock for aircraft tires were then tested for brittle point to determine effect of loss of plasticizer through migration. Essentially, the migration test involves a pressure on an assembly consisting of a sheet of experimental compound placed between two sheets of aircraft tire carcass stock and the assembly placed in an oven for 28 days at 70°C. Following this treatment, low temperature flexibility of the experimental compounds is seriously impaired and again the brittle point data for the various plasticizers is approximately equal within experimental error. On the other hand, a compound containing no plasticizer when subjected to the same treatment was improved in low temperature flexibility and after migration comes up with about the same brittle point as the compounds containing plasticizer. No explanation is offered for this unusual phenomenon.

This fact was learned just prior to the expiration of this contract and time did not allow further investigation along this line. Explanation for this improved low temperature property on unplasticized stock might be an important step toward solution of this problem. There is no apparent relationship after migration, between the amount of plasticizer retained and low temperature flexibility. This is shown in the data where with approximately the same plasticizer level the difference in brittle point before and after migration is considerable. It was also noted that the migration test resulted in considerable increase in Shore Hardness on plasticized stock. Stock without plasticizer shows no hardness change after migration treatment. No other physical properties were measured on the experimental stocks after migration.

In addition to an extensive study of various plasticizers, plasticizer combinations, etc., this program covered a study of the effect of different commercially available types of butyl rubber on low temperature brittle point, and along this line the manufacturers of butyl rubber were contacted in an effort to obtain experimental polymers. However, we were informed that no experimental polymers were available for this development.

Commercially, four grades of butyl are available, with the difference in these being in the percentage of isoprene which is copolymerized with isobutylene in preparation of the polymer. These available grades of butyl contain 1.0, 2.0, 2.5, and 3.0 per cent of isoprene. These four grades were investigated, evaluated, and compared for low temperature properties. From one of the manufacturers of butyl rubber it was learned that polymers had been prepared in which different amounts of styrene were used, and the polymers evaluated for low temperature properties. The report was that the styrene polymers were no better at low temperatures than the regular butyl which contains isoprene.

Commercial carbon blacks of different particle size, of diverse methods of manufacture, etc., were investigated to determine their effect on brittle point and on physical properties, and it was noted that brittle point on the various blacks was almost the same except for the extremely fine particle blacks, SAF and EPC, both of which imparted less desirable low temperature properties than did the coarser blacks. If maintaining highest possible tensile strength in a butyl inner tube is important, then with either SAF or EPC blacks, the black loading should be in the range of 35 parts per 100 RHC (rubber hydrocarbon). At this loading, maximum tensile values are developed and decrease with successive load increments. In this respect butyl differs with the commonly used synthetic rubbers which exhibit maximum tensile values with loadings of 60 and more of carbon black.

Whereas, the above discussed components of a butyl inner tube compound, that is, polymer, plasticizer, and carbon blacks are the major constituents in the composition, several other ingredients which are present only in smaller amount were investigated and the effect of their variation noted. In this classification, and which might be considered as of minor importance in the butyl inner tube formula, are zinc oxide, the vulcanization accelerator activator, special compounding ingredients, peroxide curing agents, and effect of high temperature processing on a butyl-carbon black mixture in a separate high temperature mixing stage, carried out prior to completion of the final mix.

In addition to the items above mentioned, the investigation included some materials which do not fall in any of the above classifications and are here listed as special materials. In this listing are the following:

1. Tellurac - R. T. Vanderbilt Co.

Tellurium diethyldithiocarbamate.

2. Polyac - E. I. du Pont de Nemours.

poly p-dinitroso benzene.

A recent communication from Esso laboratories indicated outstanding improvement in rebound, modulus and physical properties generally, by mixing and adding carbon black to butyl in the Banbury at temperatures in excess of 400°F. Plasticizers incidentally were added in a subsequent mix and at normal processing temperatures, 220 - 250°F. The improved rebound obtained through this treatment indicated a possibility for improved low temperature flexibility and, consequently, the suggested recommendations were investigated. This processing technique effected an improvement in low temperature properties on the stock before migration treatment; however, after migration low temperature brittle point was the same on the high temperature mixed stock as that of the normal mix.

The following butyl rubber inner tube compound will meet the military requirements including low temperature brittle point of -70°F.

GR-I 18	100
Zinc Oxide	5
Polyac	0.4
Philblack E or EPC Black	35 - 45
Plasticizer*	20 - 25
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2

*Any one of the plasticizers or a combination of same from the list of plasticizers with satisfactory freeze resistance.

SUMMARY AND CONCLUSIONS

Plasticizers were compounded into butyl tube stock formulations which produced satisfactory low temperature properties, that is, brittle points of -70°F. or lower. Unfortunately, in the laboratory, following migration tests used for evaluation of the experimental compounds, the desirable low temperature properties were not retained. It is not known whether the laboratory conditions as used are more or less severe than service conditions. It is recommended, therefore, that to properly evaluate some of the suggested plasticizers that inner tubes for aircraft tires be fabricated using some of the plasticizers which exhibited satisfactory low temperature flexibility and that the tubes be tested both before and after a definite number of landings when mounted in airplane tires.

Results of this work demonstrate that certain plasticizers will produce adequate low temperature flexibility according to laboratory tests; however, it was found that the plasticizers are not adequately retained when placed in contact with a natural rubber compound, or, in other words, migrate to the rubber compound.

Some of the plasticizers produced improved brittle points with increased plasticizer. The range of investigation covered 20, 25 and in some instances 30 parts plasticizer per 100 parts butyl rubber.

Butyl polymers with varying percentages of isoprene and resulting variation in degree of chemical unsaturation showed slight, if any, differences in low temperature flexibility.

An investigation of the low temperature properties of different carbon blacks showed, except for the extremely fine particle size blacks, no difference in brittle point. With EPC and SAF blacks, brittle points are not as low as with the coarser blacks. However, only these two blacks will meet the military specifications on tensile strength for compounds for inner tubes to be used in aircraft tires. With these two blacks, maximum tensile values are obtained with 35 to 40 parts black per 100 parts rubber hydrocarbon. Increased black loadings result in lower tensile strength.

No significant differences were noted with variation in zinc oxide; however, indications are that with higher amounts of zinc oxide, brittle points are poorer. Therefore, the recommended amount of zinc oxide is 5 parts per 100 parts rubber hydrocarbon. This is the conventional amount used for activation.

Of the special materials investigated, silicones, peroxide curatives, etc., no improvement was obtained in low temperature flexibility.

The more recently recommended technique for mixing butyl rubber compounds at high temperatures, in excess of 400°F., failed to show appreciable improvement in low temperature flexibility or of plasticizer retentivity in butyl rubber.

TESTING METHODS AND PROCEDURES

Preparation: A.S.T.M. D 15-50T
This procedure is followed with the exceptions here listed.

The batch is mixed in a 1100 cc. volume laboratory Banbury in which all materials are added except sulfur and accelerator. The Banbury batch is sheeted on laboratory mill, allowed from two to four hours for cooling, and sulfur and accelerator then added on 6x12 mill.

Stress Strain (Tensile) etc.: A.S.T.M. D 412-49T

Tear: Crescent Method A.S.T.M. D 624-48

Hardness: Shore Durometer Type A A.S.T.M. D 676-49T

Rebound: Goodyear-Healy A.S.T.M. D 1054-49T

Low Temperature Embrittlement: American Cyanamide - Graves Modified A.S.T.M. D 746-44T

Test piece 1.5" x 0.25" x 0.075"

Test pieces are conditioned for 2.5 minutes at the testing temperature. At the end of this period the test pieces are subjected to deflection and then examined for failure. A failure is considered to have occurred if the test piece is broken into two separate pieces. A cracked or partially broken test piece is not considered a failure.

For preliminary investigation two test pieces are used at each testing temperature. At the final temperature, 10 test pieces are used and 5 or more must pass for an OK.

Considerable controversy is encountered on the merits of various low temperature test methods. Our use of the Graves apparatus shall not be construed as approval of this method and no attempt will be made to discuss the relative merits of the various test methods and testing procedures. However, suffice it to say that the Graves apparatus is now used for evaluation of compounds for inner tubes for aircraft and for this reason was selected for determining the brittle point data shown in this report.

TR Test:

This test is used for determining the freezing point, low temperature elasticity and crystallization tendencies of rubber and rubber-like materials. Details of this test are described in India Rubber World 1951, Volume 124, Page 180.

Test piece. Constricted portion 4.0" x .075" x .075" with tabby ends 0.25" square.

Original elongation 50%
Freezing temperature -89°F.
Conditioning period 2.5 min.
Temperature rise 1.8°F.

It will be noted in the data in this report that the temperature at 40% retraction show good agreement with brittle point temperature as determined by the Graves apparatus.

Volatility Test:

A. On plasticizer itself

Heat a 10 gram sample in a glass petri dish whose diameter is approximately 2.5 inches. If available use an oven having a rotating shelf.

Heat 5 hours @ 325°F.

B. On compounded stock

Using a sample from a tensile sheet (approximately .075" gauge) cut 2" x 1" and determine loss under following conditions:

70 hours @ 212°F.
48 hours @ 300°F.

Migration Test:

Cut a piece of experimental stock from a tensile sheet (approximately .075" gauge) 2" x 1" and place between two sheets of airplane tire carcass stock of the same size, wrap the assembly in aluminum or other metallic foil and place between 8" x 10" sheets of plate glass. The load on the test pieces shall be 1 kilogram per square inch of sample area.

The assembly shall be placed in an oven at 158°F.

The assembly and the experimental stock shall be weighed at 24 hour intervals for the first four days and at weekly intervals for a period of four weeks.

A. PLASTICIZERS INCOMPATIBLE WITH BUTYL

Formula for plasticizer evaluation:

GR-I 18	100
Zinc Oxide	5
MAF Black	55
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2
Plasticizer	Variable
Curing Temperature	287°F.

TABLE I. Modulus @ 300% - Pounds per square inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall #4141	20	275	475	600	700	750
C. P. Hall #4141	25	175	325	425	500	600
Plasticizer SC	20	225	400	475	625	725
Plasticizer SC	25	200	400	450	600	700
Plast DP-200	20	125	225	275	325	325
Plast DP-200	25	150	250	325	375	450
Plast DP-520	20	NC	NC	NC	NC	NC
Plast DP-520	25	NC	NC	NC	NC	NC
Plast SC	(20)	250	425	550	650	725
& Silicone L-45	(5)					
KP-140	20	150	350	450	500	550
KP-140	25	150	350	350	450	500
P-1	20	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC
TP90B	25	150	350	450	550	650
Ucon LB-65	20	150	375	475	550	650
Ucon LB-65	25	50	325	350	450	550

TABLE II. Tensile @ Break - Pounds per square inch

C. P. Hall #4141	20	1575	1800	1750	1625	1475
C. P. Hall #4141	25	1500	1800	1600	1700	1625
Plasticizer SC	20	1575	1925	1725	1725	1675
Plasticizer SC	25	1200	1600	1600	1600	1525
Plast DP-200	20	13[illegible]5	1725	1625	1550	1525
Plast DP-200	25	1375	1750	1900	1775	1800
Plast DP-520	20	NC	NC	NC	NC	NC
Plast DP-520	25	NC	NC	NC	NC	NC
Plast SC	(20)	1600	1650	1550	1450	1500
& Silicone L-45	(5)					
KP-140	20	1500	1825	1900	1800	1700
KP-140	25	1050	1775	1800	1925	1750
P-1	20	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC
TP90B	20	1725	1950	1900	1725	1650
TP90B	25	1450	1850	1875	1800	1650
Ucon LB-65	20	1300	1850	1750	1650	1500
Ucon LB-65	25	1000	1675	1700	1600	1450

NC indicates no cure.

TABLE III. Elongation @ Break - per cent

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall #4141	20	740	690	610	565	500
C. P. Hall #4141	25	810	725	645	605	570
Plasticizer SC	20	790	710	660	600	555
Plasticizer SC	25	860	740	765	660	625
Plast DP-200	20	765	745	705	650	620
Plast DP-200	25	930	765	725	685	675
Plast DP-520	20	NC	NC	NC	NC	NC
Plast DP-520	25	NC	NC	NC	NC	NC
Plast SC & Silicone L-45	(20) (5)	755	655	610	560	530
KP-140	20	890	715	680	645	610
KP-140	25	1020	740	730	690	650
P-1	20	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC
TP90B	20	795	665	600	550	510
TP90B	25	610	655	660	615	555
Ucon LB-65	20	820	710	670	625	575
Ucon LB-65	25	1070	710	655	625	570

TABLE IV. Tear - pounds per inch

C. P. Hall #4141	20	240	239	190	168	158
C. P. Hall #4141	25	196	205	187	183	147
Plasticizer SC	20	230	248	202	193	150
Plasticizer SC	25	181	239	265	266	268
Plast. DP-200	20	212	226	200	165	144
Plast. DP-200	25	216	274	252	202	190
Plast. DP-520	20	NC	NC	NC	NC	NC
Plast. DP-520	25	NC	NC	NC	NC	NC
Plast. SC & Silicone L-45	(20) (5)	214	210	188	143	195
KP-140	20	230	232	242	182	176
KP-140	25	174	207	214	171	178
P-1	20	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC
TP90B	20	220	220	202	176	161
TP9CB	25	179	216	188	149	134
Ucon LB-65	20	158	260	196	153	134
Ucon LB-65	25	128	224	187	168	136

NC indicates no cure.

TABLE V. Hardness (Shore Durometer Type A)

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall #4141	20	38	42	44	46	48
C. P. Hall #4141	25	35	39	40	42	44
Plasticizer SC	20	36	39	41	44	46
Plasticizer SC	25	35	38	39	41	43
Plast. DP-200	20	35	40	42	44	45
Plast. DP-200	25	35	37	39	40	40
Plast. DP-520	20	NC	NC	NC	NC	NC
Plast. DP-520	25	NC	NC	NC	NC	NC
Plast. SC & Silicone L-45	(20) (5)	38	40	42	45	46
KP-140	20	35	40	42	44	44
KP-140	25	34	36	38	40	42
P-1	20	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC
TP90B	20	37	40	43	45	46
TP90B	25	34	38	40	42	44
Ucon LB-65	20	36	40	44	45	46
Ucon LB-65	25	34	39	41	43	45

NC indicates no cure.

TABLE VI. Rebound (Goodyear-Healy) 60 Min. @ 287°F.

Embrittlement (American Cyanamide-Graves) 60 Min. @ 287°F.

TR (40% Retraction--Original Elongation 50%) 60 Min. @ 287°F.

	Parts Per 100 GR-I	Rebound	Embrittlement	TR
C. P. Hall #4141	20	51%	OK @ -58°F.	-38°F.
C. P. Hall #4141	25	49.6	" -54°	-35°
Plasticizer SC	20	50.6	" -54°	-24°
Plasticizer SC	25	49.6	" -60°	-19°
Plast. DP-200	20	32.9	" -56°	-54°
Plast. DP-200	25	41.1	" -58°	-53°
Plast. DP-520	20	NC	NC	NC
Plast. DP-520	25	NC	NC	NC
Plast. SC	(20)	47.8	OK @ -54°	-42°
& Silicone L-45	(5)			
KP-140	20	37.4	-	-
KP-140	25	36.1	-	-
P-1	20	NC	NC	NC
P-1	25	NC	NC	NC
P-8	20	NC	NC	NC
P-8	25	NC	NC	NC
TP90B	20	46.4	OK @ -67°	-58°
TP90B	25	46.9	-	-
Ucon LB-65	20	41.6	OK @ -60°	-52°
Ucon LB-65	25	41.6	" -60°	-53°

NOTE:

No physical data is shown on the following plasticizers which are incompatible in butyl rubber.

a. Dibutyl maleate
b. Tributyl phosphate
c. Dibenzyl ether
d. Pycal 70 - From Atlas Powder Co.
e. Dibutyl aconitate - C. P. Hall Co.

NC indicates no cure.

TABLE VII. TR- Temperature Retraction

Cure 60 @ 287°F.

Temperature data in minus degrees Fahrenheit for percentage retractions (T-1, T-2, etc.) indicated.

	Parts Per 100 GR-I	T-1	T-2	T-3	T-5	T-10	T-20	T-30
C. P. Hall #4141	20	89°F.	87°F.	82°F.	74°F.	63°F.	53°F.	45°F.
C. P. Hall #4141	25	-	89	87	74	63	51	42
Plasticizer SC	20		89	78	67	55	40	31
Plasticizer SC	25		89	78	67	51	36	26
Plasticizer DP-200	20	89	85	83	76	71	63	58
Plasticizer DP-200	25	83	83	78	73	69	52	56
Plasticizer DP-520	20	NC	NC	NC	NC	NC	NC	NC
Plasticizer DP-520	25	NC	NC	NC	NC	NC	NC	NC
Plasticizer SC & Silicone L-45	(20) (5)	85	83	78	73	65	55	49
P-1	20	NC	NC	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC	NC	NC
TP90B	20	-	89	87	80	73	65	60
Ucon LB-65	20	-	87	83	76	70	63	58
Ucon LB-65	25	-	87	83	76	70	63	56

	Parts Per 100 GR-I	T-40	T-50	T-60	T-70	T-80	T-90
C. P. Hall #4141	20	38	31	25	15	9	+9
C. P. Hall #4141	25	36	27	18	13	8	+9
Plasticizer SC	20	24	17	11	9	1	+9
Plasticizer SC	25	19	13	10	8	+3	+14
Plasticizer DP-200	20	54	51	46	41	33	18
Plasticizer DP-200	25	53	47	42	36	27	7
Plasticizer DP-520	20	NC	NC	NC	NC	NC	NC
Plasticizer DP-520	25	NC	NC	NC	NC	NC	NC
Plasticizer SC & Silicone L-45	(20) (5)	42	36	29	20	11	0
P-1	20	NC	NC	NC	NC	NC	NC
P-1	25	NC	NC	NC	NC	NC	NC
P-8	20	NC	NC	NC	NC	NC	NC
P-8	25	NC	NC	NC	NC	NC	NC
TP90B	20	58	54	50	45	38	27
Ucon LB-65	20	52	51	46	44	36	26
Ucon LB-65	25	53	50	47	44	36	26

NOTE: No TR data obtained for KP-140 plasticizer at 20 and 25 parts loading and for TP90B at 25 parts loading.

NC indicates no cure.

B. SILICONE DC 510 FLUID AND ESTER PLASTICIZER IN BUTYL

Formula:

GR-I	100
Zinc Oxide	5
MAF black	55
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2
(DC 510 Fluid)	
(Adipol 2 EH)	25 combined total
Curing temperature	287°F.

TABLE VIII.

Modulus @ 300% - Pounds per square inch

Parts DC 510 Fluid	Parts Adipol 2 EH	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
0	25	100	525	650	800	875
5	20	175	500	600	775	850
10	15	150	500	600	775	850
15	10	100	450	550	700	750
20	5	75	350	525	625	650

Tensile @ Break - Pounds per square inch

Parts DC 510 Fluid	Parts Adipol 2 EH	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
0	25	775	1725	1900	1600	1750
5	20	1050	1825	1825	1750	1650
10	15	1150	1675	1650	1675	1450
15	10	1000	1650	1650	1500	1500
20	5	600	925	1000	1100	850

Elongation @ Break - Per cent

Parts DC 510 Fluid	Parts Adipol 2 EH	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
0	25	830	650	630	525	510
5	20	810	725	645	600	545
10	15	880	685	635	590	490
15	10	930	685	650	540	520
20	5	825	555	500	460	380

Tear - Pounds per inch

Parts DC 510 Fluid	Parts Adipol 2 EH	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
0	25	97	226	206	147	155
5	20	140	210	220	186	150
10	15	148	196	206	159	137
15	10	108	240	175	163	179
20	5	107	163	153	132	153

TABLE VIII. (Contd.)

Hardness: (Shore Durometer Type A)

Parts DC 510 Fluid	Parts Adipol 2 EH	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
0	25	34	43	45	48	49
5	20	36	43	45	47	49
10	15	38	44	46	48	49
15	10	38	46	49	51	52
20	5	37	43	45	47	48

TABLE IX. Rebound (Goodyear-Healy) 60 Min. @ 287°F.

Embrittlement (American Cyanamid-Graves) 60 Min. @ 287°F.

TR (40% Retraction - Original Elongation 50%) 60 Min. @ 287°F.

Parts DC 510 Fluid	Adipol 2 EH	Rebound	Embrittlement	TR
0	25	58.4%	OK -72°F.	-72°F.
5	20	55.4	" -72	-71
10	15	52.0	" -72	-66
15	10	45.5	" -53	-69
20	5	37.4	" -49	-55

Remarks:

DC 510 fluid was incorporated into batch by first premixing this material into dry black.

Batch with 20 parts DC 510 fluid was mixed with difficulty and batch with 25 parts was impossible to mix, and therefore no data is shown for 25 parts.

TABLE X. <u>TR - Temperature Retraction</u>

Cure 60 @ 287°F.

Temperature in minus degrees Fahrenheit for percentage retraction indicated (T-1, T-2, etc.)

Parts DC 510 Fluid	Parts Adipol 2 EH	T-1	T-2	T-3	T-5	T-10	T-20	T-30
0	25	-	-	-	88	86	82	77
5	20	-	-	-	87	83	79	73
10	15	-	-	87	84	81	74	71
15	10	-	87	85	82	78	76	74
20	5	83	82	80	76	71	63	59

Parts DC 510 Fluid	Parts Adipol 2 EH	T-40	T-50	T-60	T-70	T-80	T-90
0	25	72	71	67	63	58	46
5	20	71	67	63	60	54	44
10	15	66	63	60	56	51	38
15	10	69	63	57	53	46	31
20	5	55	52	48	44	39	26

C. PLASTICIZERS WITH DEFICIENT FREEZE RESISTANCE

The formula is the same as shown in Section A. for plasticizers incompatible with butyl.

TABLE XI. Modulus @ 300% - Pounds per square inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Forum 40 Oil	15	400	575	800	900	1000
Forum 40 Oil	20	350	575	700	850	1000
Forum 40 Oil	25	275	475	600	725	825
C. P. Hall 3890-A	20	275	500	625	775	850
C. P. Hall 3890-A & Silicone L-45	(20) (5)	325	400	575	700	775
Plast. MT-511	20	200	375	400	500	625
Plast. MT-511	25	125	275	350	475	525
VR-1 Ester	20	175	325	375	475	575
VR-1 Ester	25	125	225	325	350	400
10-A Plast.	20	150	550	725	850	950
Dibutyl Sebacate	20	200	600	700	875	950
Dibutyl Sebacate	25	150	450	600	750	825
Adipol ODY	20	200	525	650	800	875
Adipol ODY	25	200	475	600	725	775
Ohopex R-9	20	75	125	150	175	175
Ohopex R-0	25	NC	75	75	125	75
KP-555	20	375	550	675	800	675
KP-555	25	275	425	550	650	750
Monoplex S-71	20	150	250	300	350	350
Monoples S-71	25	125	175	225	250	250

NC indicates no cure.

TABLE XII. Tensile @ Break - Pounds per square inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Forum 40 Oil	15	1725	1925	1950	1950	1875
Forum 40 Oil	20	1675	1925	2000	1950	1750
Forum 40 Oil	25	1325	1875	1875	1825	1725
C. P. Hall 3890-A	20	1500	1750	1800	1625	1550
C. P. Hall 3890-A & Silicone L-45	(20) (5)	1625	1625	1625	1625	1500
Plast. MT-511	20	1525	1775	1775	1725	1600
Plast. MT-511	25	825	1275	1325	1425	1475
VR-1 Ester	20	1275	1725	1825	1900	1675
VR-1 Ester	25	1050	1775	1775	1875	1775
10-A Past.	20	1150	1825	1875	1900	1600
Dibutyl Sebacate	20	1150	1925	1800	1800	1700
Dibutyl Sebacate	25	1200	1775	1850	1675	1650
Adipol ODY	20	1450	1975	1975	1950	1800
Adipol ODY	25	1350	1950	1950	1825	1725
Ohopex R-9	20	475	1050	1325	1450	1425
Ohopex R-9	25	NC	625	725	950	725
KP-555	20	1925	1975	1950	1750	1750
KP-555	25	1775	1975	1825	1875	1750
Monoplex S-71	20	1400	1800	1700	1850	1800
Monoplex S-71	25	1300	1575	1750	1825	1775

TABLE XIII. Elongation @ Break - Per cent

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Forum 40 Oil	15	810	750	655	600	555
Forum 40 Oil	20	800	715	690	650	520
Forum 40 Oil	25	715	745	690	630	550
C. P. Hall 3890-A	20	795	645	650	545	490
C. P. Hall 3890-A & Silicone L-45	(20) (5)	765	670	630	580	540
Plast. MT-511	20	805	730	660	615	585
Plast. MT-511	25	1010	780	720	685	655
VR-1 Ester	20	930	805	780	725	700
VR-1 Ester	25	935	845	790	765	720
10-A Plast.	20	770	655	605	575	470
Dibutyl Sebacate	20	775	655	590	520	495
Dibutyl Sebacate	25	840	645	630	555	510
Adipol ODY	20	850	730	695	620	555
Adipol ODY	25	875	735	685	610	570
Ohopex R-9	20	1060	1015	1000	980	980
Ohopex R-9	25	NC	1050	900	1040	885
KP-555	20	815	720	680	580	550
KP-555	25	825	755	680	645	580
Monoplex S-71	20	895	895	805	800	780
Monoplex S-71	25	940	875	885	845	835

NC indicates no cure.

TABLE XIV   Tear - Pounds per inch

	Parts Per 100 GR-1	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Forum 40 Oil	15	224	238	232	212	198
Forum 40 Oil	20	204	216	210	202	174
Forum 40 Oil	25	189	222	214	182	177
C. P. Hall 3890-A	20	216	224	219	196	157
C. P. Hall 3890-A & Silicone L-45	(20) (5)	245	214	190	160	210
Plast. MT-511	20	197	237	191	178	181
Plast. MT-511	25	167	230	256	245	297
VR-1 Ester	20	185	256	257	267	245
VR-1 Ester	25	172	245	242	232	210
10-A Plast.	20	164	214	188	173	189
Dibutyl Sebacate	20	151	214	204	190	142
Dibutyl Sebacate	25	139	202	194	159	156
Adipol ODY	20	210	254	228	200	159
Adipol ODY	25	199	236	240	232	156
Ohopex R-9	20	79	148	180	191	208
Ohopex R-9	25	NC	100	112	135	137
KP-555	20	244	246	231	175	169
KP-555	25	246	230	185	203	204
Monoplex S-71	20	206	216	240	236	230
Monoplex S-71	25	174	228	222	240	218

TABLE XV.   Hardness (Shore Durometer Type A)

Forum 40 Oil	15	43	47	49	51	52
Forum 40 Oil	20	41	45	47	49	51
Forum 40 Oil	25	37	42	44	46	47
C. P. Hall 3890-A	20	38	44	47	49	50
C. P. Hall 3890-A & Silicone L-45	(20) (5)	36	43	45	47	48
Plast. MT-511	20	42	45	47	48	49
Plast. MT-511	25	40	44	45	46	46
VR-1 Ester	20	38	41	43	45	46
VR-1 Ester	25	33	38	40	41	43
10-A Plast.	20	38	44	46	48	50
Dibutyl Sebacate	20	38	45	47	49	50
Dibutyl Sebacate	25	34	41	44	46	47
Adipol ODY	20	37	44	46	49	51
Adipol ODY	25	35	42	45	47	49
Ohopex R-9	20	30	34	36	37	38
Ohopex R-9	25	25	30	32	33	33
KP-555	20	42	46	47	51	52
KP-555	25	39	44	46	48	49
Monoplex S-71	20	37	39	40	41	41
Monoplex S-71	25	32	36	38	39	39

NC indicates no cure.

TABLE XVI.

Rebound (Goodyear-Healy) 60 Min. @ 287°F.

Embrittlement (American Cyanamide-Graves) 60 Min. @ 287°F.

TR (40% Retraction - Original Elongation 50%) 60 Min. @ 287°F.

	Parts Per 100 GR-I	Rebound	Embrittlement	TR
Forum 40 Oil	15	45.1%	OK @ -54°F.	-57°F.
Forum 40 Oil	20	46.4	-62	-58
Forum 40 Oil	25	50.1	-62	-61
C. P. Hall 3890-A	20	56.4	-54	-67
C. P. Hall 3890-A & Silicone L-45	(20) (5)	56.4	-60	-67
Plast. MT-511	20	35.3	-44	-45
Plast. MT-511	25	32.9	-44	-45
VR-1 Ester	20	43.7	-49	-54
VR-1 Ester	25	44.6	-49	-56
10-A Plast.	20	54.9	-54	-54
Dibutyl Sebacate	20	59.5	-54	-47
Dibutyl Sebacate	25	61.5	-54	-42
Adipol ODY	20	61.5	" -56	-47
Adipol ODY	25	64.1	" -56	-49
Ohopex R-9	20	44.2	" -56	-49
Ohopex R-9	25	43.3	" -67	-45
KP-555	20	54.9	" -53	-59
KP-555	25	59.0	" -56	-60
Monoplex S-71	20	45.1	" -48	-59
Monoplex S-71	25	48.2	" -58	-58

TABLE XVII. <u>TR - Temperature Retraction</u>

Temperature in minus degrees Fahrenheit for percentage retraction indicated (T-1, T-2, etc.)

	Parts Per 100 GR-I	T-1	T-2	T-3	T-5	T-10	T-20	T-30
Forum 40 Oil	15	89	87	83	81	73	66	62
Forum 40 Oil	20	87	85	83	81	74	67	62
Forum 40 Oil	25	87	85	83	82	76	68	65
C. P. Hall 3890-A	20	-	-	89	88	87	76	71
C. P. Hall 3890-A & Silicone L-45	(20) (5)	-	-	89	88	83	76	71
Plasticizer MT-511	20	82	72	70	64	60	53	49
Plasticizer MT-511	25	87	72	70	63	60	53	48
VR-I Ester	20	-	-	89	82	73	65	58
VR-I Ester	25	-	-	89	85	77	66	60
10-A Plasticizer	20	83	82	81	73	71	62	56
Dibutyl Sebacate	20	-	87	83	80	69	60	54
Dibutyl Sebacate	25	-	89	87	80	67	55	47
Adipol ODY	20	-	-	87	83	73	61	53
Adipol ODY	25	83	-	77	71	60	47	39
Ohopex R-9	20	-	87	86	83	74	62	51
Ohopex R-9	25	-	-	87	84	76	65	55
KP-555	20	87	86	84	80	74	67	63
KP-555	25	87	85	83	80	74	68	63
Monoplex S-71	20	-	87	85	83	78	71	64
Monoplex S-71	25	-	-	87	86	81	73	66
No Plasticizer	(None)	82	81	77	71	64	56	51

	Parts Per 100 GR-I	T-40	T-50	T-60	T-70	T-80	T-90
Forum 40 Oil	15	57	54	40	44	36	18
Forum 40 Oil	20	58	54	51	45	37	22
Forum 40 Oil	25	61	57	54	48	40	28
C. P. Hall 3890-A	20	67	63	60	56	49	38
C. P. Hall 3890-A & Silicone L-45	(20) (5)	67	63	60	56	49	38
Plasticizer MT-511	20	45	40	36	29	17	4
Plasticizer MT-511	25	45	39	34	26	19	0
VR-I Ester	20	54	51	46	42	35	19
VR-I Ester	25	56	51	47	43	35	15
10-A Plasticizer	20	54	51	46	44	37	27
Dibutyl Sebacate	20	47	42	36	27	19	4
Dibutyl Sebacate	25	42	36	30	23	13	0
Adipol ODY	20	47	42	35	29	21	20
Adipol ODY	25	31	28	22	13	4	+10
Ohopex R-9	20	43	32	22	11	+ 1	+23
Ohopex R-9	25	45	31	12	+ 1	+18	+34
KP-555	20	59	55	52	48	42	32
KP-555	25	60	56	53	50	45	35
Monoplex S-71	20	59	52	43	27	13	+ 1
Monoplex S-71	25	58	47	35	22	9	+ 7
No Plasticizer	(None)	45	41	36	31	26	9

TABLE XVIII. TR - Temperature Retraction After 4 Weeks Migration at 158°F.

	Parts Per 100 GR-I Original	After Migration	T-1	T-2	T-3	T-5	T-10	T-20	T-30
Forum 40 Oil	15	9.5	87	85	83	78	72	64	59
Forum 40 Oil	20	11.6	87	85	83	79	72	64	60
Forum 40 Oil	25	14.0	87	85	83	79	72	65	60
No Plasticizer	None	None	88	86	83	78	71	62	56

	Parts Per 100 GR-I Original	After Migration	T-40	T-50	T-60	T-70	T-80	T-90
Forum 40 Oil	15	9.5	55	51	46	42	36	18
Forum 40 Oil	20	11.6	56	52	47	43	36	21
Forum 40 Oil	25	14.0	56	52	46	41	33	17
No Plasticizer	None	None	54	48	45	40	34	24

D. PLASTICIZERS WITH SATISFACTORY FREEZE RESISTANCE

The formula is the same as shown in Section A. for plasticizers incompatible with butyl.

TABLE XIX. Modulus @ 300% - Pounds per square inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall 3890-A	25	175	400	525	600	700
Adipol 2 EH	20	160	540	680	825	910
Adipol 2 EH	25	100	525	650	800	875
10-A Plast.	25	NC	500	650	775	850
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	150	450	525	675	775
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	150	500	560	750	825
Di 2 Ethyl Hexyl Ether	20	225	425	575	700	850
Di 2 Ethyl Hexyl Ether	25	275	400	550	650	725
Trioctyl Phosphate	20	150	500	600	650	750
Trioctyl Phosphate	25	75	375	500	550	625
Butyl Cellosolve Pelargonate	20	350	600	725	875	950
Butyl Cellosolve Pelargonate	25	225	450	600	725	850
Butyl Carbitol Pelargonate	20	175	425	550	675	750
Butyl Carbitol Pelargonate	25	175	375	500	600	725
Diisobutyl Azelate	20	225	525	675	850	975
Diisobutyl Azelate	25	200	500	650	800	875
Monoplex DOS	20	225	525	675	825	900
Monoplex DOS	25	225	450	575	675	750
Hexyl Ether	20	450	650	800	950	1000
Hexyl Ether	25	300	475	575	750	800

NC indicates no cure.

TABLE XX. Tensile at Break - Pounds per square inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall 3890-A	25	1250	1775	1700	1650	1625
Adipol 2 EH	20	1070	1910	1870	1750	1650
Adipol 2 EH	25	775	1725	1900	1600	1750
10-A Plast.	25	NC	1900	1725	1875	1750
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	1150	1875	1900	1875	1650
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	1000	1900	1775	1650	1700
Di 2 Ethyl Hexyl Ether	20	1400	1800	1825	1925	1775
Di 2 Ethyl Hexyl Ether	25	1625	1775	1800	1700	1800
Trioctyl Phosphate	20	1325	2075	2075	1950	1800
Trioctyl Phosphate	25	1000	1875	2050	1900	1850
Butyl Cellosolve Pelargonate	20	1625	1950	1850	1750	1625
Butyl Cellosolve Pelargonate	25	1625	1850	1825	1775	1600
Butyl Carbitol Pelargonate	20	1300	1950	1925	1775	1600
Butyl Carbitol Pelargonate	25	1425	2000	2000	1950	1700
Diisobutyl Azelate	20	1500	1800	1900	1800	1650
Diisobutyl Azelate	25	1225	1875	1950	1825	1850
Monoplex DOS	20	1325	1925	1975	1950	1925
Monoplex DOS	25	825	2000	1875	1875	1900
Hexyl Ether	20	1675	1750	1750	1625	1550
Hexyl Ether	25	1500	1675	1775	1600	1425

TABLE XXI. Elongation @ Break - Per cent

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall 3890-A	25	810	670	635	590	550
Adipol 2 EH	20	800	680	640	565	515
Adipol 2 EH	25	830	650	630	525	510
10-A Plast.	25	NC	680	615	595	530
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	810	710	665	600	500
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	895	695	635	540	550
Di 2 Ethyl Hexyl Ether	20	875	755	705	700	595
Di 2 Ethyl Hexyl Ether	25	800	775	715	645	640
Trioctyl Phosphate	20	955	765	705	670	600
Trioctyl Phosphate	25	945	745	740	675	635
Butyl Cellosolve Pelargonate	20	755	745	655	575	500
Butyl Cellosolve Pelargonate	25	870	730	655	610	540
Butyl Carbitol Pelargonate	20	800	750	685	620	505
Butyl Carbitol Pelargonate	25	840	775	725	690	570
Diisobutyl Azelate	20	900	695	680	600	495
Diisobutyl Azelate	25	785	735	700	625	585
Monoplex DOS	20	910	770	720	650	615
Monoplex DOS	25	680	815	725	680	660
Hexyl Ether	20	745	605	610	475	455
Hexyl Ether	25	800	700	680	565	490

NC indicates no cure.

TABLE XXII. Tear - Pounds per inch

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall 3890-A	25	172	218	191	200	164
Adipol 2 EH	20	150	236	200	174	169
Adipol 2 EH	25	97	226	206	147	155
10-A Plast.	25	NC	274	202	178	160
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	159	220	206	182	153
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	130	258	199	189	171
Di 2 Ethyl Hexyl Ether	20	194	208	224	180	170
Di 2 Ethyl Hexyl Ether	25	220	218	189	178	158
Trioctyl Phosphate	20	204	280	226	220	194
Trioctyl Phosphate	25	109	224	218	195	189
Butyl Cellosolve Pelargonate	20	264	238	192	169	159
Butyl Cellosolve Pelargonate	25	188	218	179	168	145
Butyl Carbitol Pelargonate	20	250	238	195	216	150
Butyl Carbitol Pelargonate	25	202	222	212	208	178
Diisobutyl Azelate	20	194	234	236	189	189
Diisobutyl Azelate	25	222	260	208	190	206
Monoplex DOS	20	202	238	214	188	182
Monoplex DOS	25	173	224	194	189	204
Hexyl Ether	20	234	204	186	172	151
Hexyl Ether	25	226	230	192	155	144

TABLE XXIII. Hardness (Shore Durometer Type A)

	Parts Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
C. P. Hall 3890-A	25	34	40	44	46	47
Adipol 2 EH	20	37	44	47	49	51
Adipol 2 EH	25	34	43	45	48	49
10-A Plast.	25	NC	42	45	48	49
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	35	41	44	46	47
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	35	41	44	46	47
Di 2 Ethyl Hexyl Ether	20	39	43	45	47	48
Di 2 Ethyl Hexyl Ether	25	39	42	44	46	48
Trioctyl Phosphate	20	35	44	45	46	48
Trioctyl Phosphate	25	32	41	43	44	44
Butyl Cellosolve Pelargonate	20	40	45	48	51	52
Butyl Cellosolve Pelargonate	25	36	42	45	48	49
Butyl Carbitol Pelargonate	20	38	44	46	48	49
Butyl Carbitol Pelargonate	25	34	40	44	46	48
Diisobutyl Azelate	20	39	45	48	50	52
Diisobutyl Azelate	25	35	43	45	47	49
Monoplex DOS	20	39	44	46	49	50
Monoplex DOS	25	35	41	44	46	47
Hexyl Ether	20	42	47	50	52	53
Hexyl Ether	25	39	42	45	47	49

NC indicates no cure.

TABLE XXIV.

Rebound (Goodyear-Healy) 60 Min. @ 287°F.

Embrittlement (American Cyanamid-Graves) 60 Min. @ 287°F.

TR (40% Retraction - Original Elongation 50%) 60 Min. @ 287°F.

	Parts Per 100 GR-I	Rebound	Embrittlement	TR
C. P. Hall 3890-A	25	58.4%	OK @ -72°F	-70°F.
Adipol 2 EH	20	58.4	-71	-71
Adipol 2 EH	25	58.4	-72	-72
10-A Plast.	25	57.4	-72	-74
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	53.9	-72	-71
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	50.1	-72	-67
Di 2 Ethyl Hexyl Ether	20	57.4%	OK @ -78°F.	-76°F.
Di 2 Ethyl Hexyl Ether	25	60.0	" -76	-74
Trioctyl Phosphate	20	51.5	" -63	-70
Trioctyl Phosphate	25	57.9	" -76	-72
Butyl Cellosolve Pelargonate	20	64.1	" -72	-74
Butyl Cellosolve Pelargonate	25	66.8	" -78	-77
Butyl Carbitol Pelargonate	20	61.5	" -72	-67
Butyl Carbitol Pelargonate	25	61.0	" -76	-67
Diisobutyl Azelate	20	57.9	" -72	-66
Diisobutyl Azelate	25	61.0	" -72	-68
Monoplex DOS	20	54.9	" -67	-70
Monoplex DOS	25	56.9	" -72	-73
Hexyl Ether	20	64.1	" -71	-72
Hexyl Ether	25	64.1	" -74	-72

TABLE XXV. TR - Temperature Retraction

Temperature in minus degrees Fahrenheit for percentage retraction indicated (T-3, T-5, etc.)

	Parts Per 100 GR-I	T-3	T-5	T-10	T-20	T-30
C. P. Hall 3890-A	25	-	89	88	81	72
Adipol 2 EH	20	89	88	82	78	75
Adipol 2 EH	25	89	88	82	79	76
10-A Plasticizer	25	-	89	87	82	78
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	89	87	83	78	74
C. P. Hall 3890A & L-41 Silicone	(15) (10)	89	87	83	74	69
Di 2 Ethyl Hexyl Ether	20	-	-	87	84	81
Di 2 Ethyl Hexyl Ether	25	-	-	85	82	79
Trioctyl Phosphate	20	-	-	83	78	74
Trioctyl Phosphate	25	-	-	80	78	76
Butyl Cellosolve Pelargonate	20	-	87	86	82	78
Butyl Cellosolve Pelargonate	25	-	-	87	83	81
Butyl Carbitol Pelargonate	20	-	85	83	77	72
Butyl Carbitol Pelargonate	25	-	85	83	77	72
Diisobutyl Azelate	20	-	85	83	77	72
Diisobutyl Azelate	25	-	87	83	78	72
Monoplex DOS	20	-	87	84	79	74
Monoplex DOS	25	-	87	85	82	77
Hexyl Ether	20	-	-	86	82	76
Hexyl Ether	25	-	-	86	82	76

	Parts Per 100 GR-I	T-40	T-50	T-60	T-70	T-80	T-90
C. P. Hall 3890-A	25	70	66	63	59	53	42
Adipol 2 EH	20	71	69	63	62	54	44
Adipol 2 EH	25	72	69	65	62	58	47
10-A Plasticizer	25	74	71	67	62	57	45
C. P. Hall 3890-A & L-41 Silicone	(20) (5)	71	67	62	57	53	38
C. P. Hall 3890-A & L-41 Silicone	(15) (10)	67	66	60	56	49	38
Di 2 Ethyl Hexyl Ether	20	76	72	68	63	56	40
Di 2 Ethyl Hexyl Ether	25	74	71	67	59	51	35
Trioctyl Phosphate	20	70	67	63	59	54	42
Trioctyl Phosphate	25	72	70	66	63	55	45
Butyl Cellosolve Pelargonate	20	74	72	68	64	58	45
Butyl Cellosolve Pelargonate	25	77	73	70	65	60	45
Butyl Carbitol Pelargonate	20	67	65	59	54	49	40
Butyl Carbitol Pelargonate	25	67	65	59	55	49	38
Diisobutyl Azelate	20	66	65	60	56	51	41
Diisobutyl Azelate	25	68	65	61	57	51	40
Monoplex DOS	20	70	66	63	58	53	41
Monoplex DOS	25	73	70	66	62	55	43
Hexyl Ether	20	72	68	64	60	53	37
Hexyl Ether	25	72	67	63	58	50	32

TABLE XXVI. TR - Temperature Retraction After 4 Weeks Migration at 158°F.

	Parts Per 100 GR-I							
	Original*	After Migration**	T-2	T-3	T-5	T-10	T-20	T-30
C. P. Hall 3890-A	25	9.6	87	85	83	75	68	63
Adipol 2 EH	20	8.8	87	84	80	74	67	62
Adipol 2 EH	25	10.9	87	86	83	74	67	63
10-A Plasticizer	25	10.7	-	86	83	75	67	63
Di 2 Ethyl Hexyl Ether	20	14.0	86	-	81	74	66	63
Di 2 Ethyl Hexyl Ether	25	18.2	87	-	80	72	64	58
Trioctyl Phosphate	20	6.6	83	-	80	72	65	60
Trioctyl Phosphate	25	7.7	83	-	80	72	66	61
Butyl Cellosolve Pelargonate	20	12.2	85	-	81	74	65	60
Butyl Cellosolve Pelargonate	25	18.9	86	-	83	73	63	58
Butyl Carbitol Pelargonate	20	9.9	85	-	80	72	65	60
Butyl Carbitol Pelargonate	25	11.8	86	-	83	75	69	63
Diisobutyl Azelate	20	8.7	87	-	85	79	71	67
Diisobutyl Azelate	25	10.8	86	-	83	77	70	63
Monoplex DOS	20	11.0	85	-	83	76	69	64
Monoplex DOS	25	4.5	85	-	83	76	69	64
Hexyl Ether	20	18.2	82	-	80	72	63	59
Hexyl Ether	25	21.6	87	-	83	81	68	62
Natural Rubber Tube Compound	4.0	4.0	83	81	80	78	75	72

NOTE:

* Parts per 100 GR-I original. This refers to the parts plasticizer per 100 parts butyl hydrocarbon which was added to the compound.

** Parts per 100 GR-I - after migration. Based on the weight loss of the sample during migration and assuming that the entire weight change is due to loss of plasticizer, this value is determined by subtracting the amount lost from the original and represents the amount of plasticizer retained after migration and is expressed in parts plasticizer per 100 parts butyl hydrocarbon.

TABLE XXVI. (Contd.)

Parts Per 100 GR-I

	Original*	After Migration**	T-40	T-50	T-60	T-70	T-80	T-90
C. P. Hall 3890-A	25	9.6	58	54	51	46	40	29
Adipol 2 EH	20	8.8	58	54	51	46	42	32
Adipol 2 EH	25	10.9	58	54	51	45	39	27
10-A Plasticizer	25	10.7	58	54	51	46	38	27
Di 2 Ethyl Hexyl Ether	20	14.0	56	54	49	44	38	26
Di 2 Ethyl Hexyl Ether	25	18.2	53	48	44	31	29	13
Trioctyl Phosphate	20	6.6	56	53	48	40	38	35
Trioctyl Phosphate	25	7.7	56	52	48	40	36	20
Butyl Cellosolve Pelargonate	20	12.2	56	52	47	45	38	24
Butyl Cellosolve Pelargonate	25	18.9	56	52	47	45	38	24
Butyl Carbitol Pelargonate	20	9.9	58	53	49	45	42	29
Butyl Carbitol Pelargonate	25	11.8	60	56	53	47	40	24
Diisobutyl Azelate	20	8.7	62	58	54	49	44	29
Diisobutyl Azelate	25	10.8	60	57	53	47	42	27
Monoplex DOS	20	11.0	60	55	52	47	41	26
Monoplex DOS	25	4.5	60	54	50	45	38	23
Hexyl Ether	20	18.2	54	49	46	41	35	22
Hexyl Ether	25	21.6	56	53	49	44	37	28
Natural Rubber Tube Compound	4.0	4.0	71	68				38

NOTE:

* Parts per 100 GR-I original. This refers to the parts plasticizer per 100 parts butyl hydrocarbon which was added to the compound.

** Parts per 100 GR-I - after migration. Based on the weight loss of the sample during migration and assuming that the entire weight change is due to loss of plasticizer, this value is determined by subtracting the amount lost from the original and represents the amount of plasticizer retained after migration and is expressed in parts plasticizer per 100 parts butyl hydrocarbon.

E. HIGH PLASTICIZER HIGH CARBON BLACK

Formula

GR-I 18	100
Zinc Oxide	5
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2
MAF Black*	Variable
Plasticizer**	Variable
Curing temperature	287°F

*MAF Black Philblack A
**Plasticizers

a. C. P. Hall's 3890-A
b. Ohio Apex 10-A
c. Ohio Apex Adipol 2 EH

TABLE XXVII.

Modulus @ 300% - Pounds per square inch

Parts Plasticizer	Parts MAF Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Plasticizer - C. P. Hall's 3890-A						
25	55	175	350	450	650	725
30	55	100	350	400	550	650
30	60	75	350	450	600	700
Ohio Apex 10-A Plasticizer						
25	55	75	400	550	725	775
30	55	50	375	475	625	700
30	60	175	425	575	675	750
Ohio Apex Adipol 2 EH						
25	55	175	425	625	750	800
30	55	150	350	525	650	750
30	60	175	425	550	650	750

TABLE XXVIII.

Tensile-@ Break - Pounds per-square inch

Parts Plasticizer	Parts MAF Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Plasticizer - C. P. Hall's 3890-A						
25	55	1250	1900	1875	1825	1750
30	55	1000	1900	1850	1775	1750
30	60	700	1800	1925	1825	1650
Ohio Apex 10-A Plasticizer						
25	55	750	2100	1950	1825	1725
30	55	375	1875	1975	1825	1700
30	60	1375	1775	1900	1775	1575
Ohio Apex Adipol 2 EH						
25	55	1250	1825	1975	1750	1600
30	55	1200	1875	1875	1700	1850
30	60	1400	1800	1750	1700	1650

TABLE XXVIX.

Elongation @ Break - Per cent

Parts Plasticizer	Parts MAF Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Plasticizer - C. P. Hall's 3890-A						
25	55	810	770	725	650	605
30	55	800	775	730	665	630
30	60	1025	780	760	670	585
Ohio Apex - 10-A Plasticizer						
25	55	1050	800	730	620	570
30	55	1050/*	780	740	640	600
30	60	900	730	715	640	565
Ohio Apex Adipol 2 EH						
25	55	825	745	710	600	535
30	55	870	775	685	615	620
30	60	880	740	660	610	565

*This elongation value beyond the limits of the machine.

TABLE XXX.

Tear - Pounds per inch

Parts Plasticizer	Parts MAF Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Plasticizer - C. P. Hall's 3890-A						
25	55	162	216	214	167	152
30	55	103	190	182	149	180
30	60	99	246	224	182	181
Ohio Apex 10-A Plasticizer						
25	55	105	228	240	190	167
30	55	59	242	236	179	169
30	60	175	264	230	212	183
Ohio Apex Adipol 2 EH						
25	55	175	222	194	183	178
30	55	130	230	188	169	151
30	60	170	236	208	228	182

TABLE XXXI.

Hardness (Shore Durometer Type A)

Plasticizer - C. P. Hall's 3890-A						
25	55	35	40	44	46	48
30	55	33	40	42	44	45
30	60	32	40	42	45	47
Ohio Apex 10-A Plasticizer						
25	55	33	41	44	47	49
30	55	29	39	42	44	46
30	60	35	41	45	47	48
Ohio Apex Adipol 2 EH						
25	55	36	42	45	47	49
30	55	33	39	44	45	46
30	60	35	41	45	46	48

TABLE XXXII.

Rebound (Goodyear-Heady) 60 Min. @ 287°F.

Embrittlement (American Cyanimid-Graves) 60 Min. @ 287°F.

TR (40% Retraction - Original Elongation 50%) 60 @ 287°F.

Parts Plasticizer	Parts MAF Black	Rebound	Embrittlement	TR
Plasticizer-C. P. Hall's 3890-A				
25	55	56.9%	OK @ -71°F.	-72°F.
30	55	59.0	" -74	-73
30	60	58.4	" -71	-72
Ohio Apex 10-A Plasticizer				
25	55	60.0	" -74	-71
30	55	61.5	" -74	-72
30	60	61.5	" -76	-73
Ohio Apex Adipol 2 EH				
25	55	61.5	" -76	-74
30	55	63.6	" -80	-76
30	60	60.5	" -80	-75

Remarks: In some cases high plasticizer and high black with a high plasticity elastomer results in a compound with improved low temperature properties.

TABLE XXXIII. TR - Temperature Retraction Data

Temperature in minus degrees Fahrenheit for percentage retraction indicated (T-5, T-10, etc.)

Parts Per 100 GR-I

Plasticizer	MAF Black	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
				Plasticizer C. P. Hall's 3890-A								
25	55	-	87	85	80	75	72	69	65	62	55	43
30	55	-	87	85	81	76	73	70	67	62	55	42
30	60	-	87	85	80	76	72	69	66	62	55	42
				Ohio Apex 10-A Plasticizer								
25	55	-	87	84	79	75	71	69	65	62	56	45
30	55	-	87	84	80	76	72	70	66	62	55	42
30	60	0	87	85	81	77	73	70	67	62	56	43
				Ohio Apex Adipol 2 EH								
25	55	-	88	86	81	77	74	71	67	63	58	46
30	55	-	88	87	82	79	76	72	69	65	60	48
30	60	-	88	87	82	78	75	72	68	64	59	47

TR - Temperature Retraction After 4 Weeks Migration At 158°F.

Plasticizer	MAF Black	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
				Plasticizer C. P. Hall's 3890-A								
30	55	86	82	75	67	62	58	54	49	45	38	20
				Ohio Apex 10-A Plasticizer								
30	55	86	81	74	66	63	56	54	49	44	38	20
				Ohio Apex Adipol 2 EH								
30	55	86	83	76	67	63	58	52	49	45	40	24

F. SPECIAL ACTIVATING AGENTS - POLYAC AND TELLURAC

Formula

	Polyac	Tellurac
GR-I 18	100.	100.
Zinc Oxide	5.	5.
MAF Black	55.	55.
Captax/Tuads (1:2 Blend)	1.54	1.54
Sulfur	2.	2.
Adipol 2 EH	20.	20.
Polyac	.4	-
Tellurac	-	.5
	183.94	184.04

Description of Materials

Polyac - E. I. du Pont

25% poly-dinitroso benzene
75% inert material

Tellurac - R. T. Vanderbilt

Telluriumdiethyldithiocarbamate

Processing

Polyac was added to batch in Banbury mixer and batch discharged after temperature reached 300°F.

Tellurac is added in final mix on 6x12 mill along with sulfur and accelerator.

TABLE XXXIV.

	10 Min.	20 Min.	30 Min.	45. Min.	60 Min.
Modulus at 300% - Pounds per square inch					
Polyac	250	900	1050	1175	1275
Tellurac	275	575	700	875	925
Tensile @ Break - Pounds per square inch					
Polyac	1100	1875	1800	1750	1700
Tellurac	1550	1975	1725	1525	1400
Elongation @ Break - Per Cent					
Polyac	760	600	555	470	430
Tellurac	830	740	630	505	455
Tear - Pounds per inch					
Polyac	131	250	201	153	149
Tellurac	195	223	154	169	152
Durometer					
Polyac	36	44	47	49	51
Tellurac	39	45	48	50	52

	Rebound	Embrittlement	TR
Polyac	61.0%	OK @ -71°F.	-70°F.
Tellurac	61.5	-71	-69

TABLE XXXV. TR - Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions T-5, T-10, etc.) indicated.

	Polyac	Tellurac
T-5	87	85
T-10	83	83
T-20	78	77
T-30	74	72
T-40	70	69
T-50	67	67
T-60	63	63
T-70	60	60
T-80	56	55
T-90	47	48

G. COMMERCIAL CARBON BLACKS IN BUTYL

Formula

GR-I 18	100.
Zinc Oxide	5.
Carbon Black	55.
Captax/Tuads (1:2 Blend)	1.54*
Sulfur	2.
Adipol 2 EH	20.
	183.54

Curing temperature 287°F.

* Accelerator used with EPC black 2.2 parts.

TABLE XXXVI.

Black	10. Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch					
Thermax	NC	150	175	250	250
P-33	75	100	125	175	150
SRF	75	275	325	475	500
MAF	160	540	680	825	910
HAF	225	475	625	825	950
EPC*	125	350	470	590	690
SAF	160	478	650	820	1000
Tensile @ Break - Pounds per square inch					
Thermax	NC	2450	2500	2375	1750
P-33	800	1425	1775	1675	1875
SRF	325	1900	2250	2225	1925
MAF	[illegible]	1910	1870	1750	1650
HAF	1825	2225	2275	2200	2175
EPC*	1275	2540	2510	2520	2570
SAF	1500	2490	2500	2400	2350
Elongation @ Break - Per cent					
Thermax	NC	880	855	745	690
P-33	830	755	755	715	700
[illegible]	1045	775	790	670	660
HAF	945	780	715	625	570
EPC*	1040	870	780	730	695
SAF	1020	820	725	645	590

* On account of retardation to cure of EPC black, accelerator for this black is 2.2 parts instead of 1.54 parts as on all of the other blacks.

NC indicates no cure.

TABLE XXXVI. (Contd.)

Black	10 Mi-.	20 Min.	30 Min.	45 Min.	60 Min.
Tear - Pounds per inch					
Thermax	NC	177	133	51	41
P-33	188	152	119	89	53
SRF	63	187	125	97	107
MAF	150	236	200	174	169
HAF	280	320	280	243	233
EPC*	196	325	306	295	295
SAF	232	346	342	310	296
Hardness (Shore Durometer Type A)					
Thermax	21	31	35	36	37
P-33	32	36	38	39	40
SRF	30	38	41	43	45
MAF	37	44	47	49	51
HAF	37	43	46	48	49
EPC*	33	42	44	46	47
SAF	37	44	47	49	51

TABLE XXXVII. Rebound (Goodyear-Healy) 60 @ 287°F.
Embrittlement (American Cyanamid-Graves) 60 @ 287°F.
TR (40% Retraction--Original Elongation 50%) 60 @ 287°F.

Black	Rebound	Embrittlement	TR
Thermax	63.6%	OK @ -65°F.	-77°F.
P-33	64.1	" -69	-74
SRF	64.6	" -67	-71
MAF	58.4	" -71	-69
HAF	55.9	" -71	-69
EPC*	53.7	" -62	-69
SAF	46.0	" -60	-68

* On account of retardation to cure of EPC black, accelerator for this black is 2.2 parts instead of 1.54 parts as on all of the other blacks.

Blacks used for this program

SRF	Witco SRF
MAF	Philblack A
HAF	Philblack O
EPC	Sid Richardson's Texas E
SAF	Philblack E

TABLE XXXVIII. TR - Temperature Retraction

Temperature data in minus degrees Fahrenheit for percentage retractions (T-3, T-5, etc.) indicated.

Black	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
Thermax	-	87	85	81	80	77	75	72	72	66	57
P-33	87	86	83	80	78	74	72	71	65	60	47
SRF	-	87	84	79	74	71	71	69	63	60	49
MAF	-	87	82	78	72	69	68	63	60	54	45
HAF	-	87	83	77	72	69	65	61	56	50	36
EPC	-	87	81	76	72	69	65	62	57	51	37
SAF	-	87	83	76	72	68	63	60	54	49	35

H. VARIABLE CARBON BLACK LOADING IN BUTYL

Formula

GR-I 18	100.
Zinc Oxide	5.
Carbon Black	Variable
Captax/Tuads (1:2 Blend)	1.54*
Sulfur	2.
Adipol 2 EH	20.

* Account retarding effect of EPC black the amount of accelerator is increased and is as follows:

Parts EPC Black	Accelerator
55	2.20
45	2.08
35	1.96

TABLE XXXIX.

Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch					
MAF 55	160	540	680	825	910
" 45	150	450	575	700	800
" 35	75	275	325	425	500
EPC*55	125	350	470	590	690
" 45	100	350	400	525	600
" 35	75	250	300	350	425
SAF 55	160	475	650	820	1000
" 45	125	400	500	700	775
" 35	75	300	375	475	600
Tensile @ Break - Pounds per square inch					
MAF 55	1070	1910	1870	1750	1650
" 45	1250	2200	2250	2000	1850
" 35	375	1975	2325	2175	1775
EPC*55	1275	2540	2510	2520	2570
" 45	1175	2700	2725	2725	2675
" 35	675	2900	2875	2675	2625
SAF 55	1500	2490	2500	2400	2350
" 45	1575	2800	2875	2775	2825
" 35	825	2875	2925	2775	2625
Elongation @ Break - Per cent-					
MAF 55	800	680	640	565	515
" 45	980	765	700	610	555
" 35	960	800	790	745	640
EPC*55	1040	870	780	730	695
" 45	1070	845	765	700	670
" 35	1105	955	875	785	740
SAF 55	1020	820	725	645	590
" 45	845	810	775	675	660
" 35	835	825	785	700	650

* See Page 42

TABLE XL.

Black	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Tear - Pounds per inch					
MAF 55	150	236	200	174	169
" 45	146	216	188	152	146
" 35	88	194	112	111	88
EPC*55	196	325	306	295	295
" 45	129	282	278	230	204
" 35	75	240	264	163	113
SAF 55	232	346	342	310	296
" 45	290	358	334	296	282
" 35	138	283	224	224	215
Hardness (Shore Durometer Type A)					
MAF 55	37	44	47	49	51
" 45	34	40	44	46	48
" 35	30	38	40	42	43
EPC*55	33	42	44	46	47
" 45	30	39	41	43	45
" 35	29	37	29	41	42
SAF 55	37	44	47	49	51
" 45	35	41	43	45	47
" 35	30	38	40	41	42

TABLE XLI.

Rebound (Goodyear-Healy) 60 Min. @ 287°F.
Embrittlement (American Cyanamid-Graves) 60 @ 287°F.
TR (40% Retraction-Original Elongation 50%) 60 @ 287°F.

Black	Rebound	Embrittlement	TR
MAF 55	58.4%	OK @ -71°C.	-70°C.
" 45	59.0	" -71	-71
" 35	62.0	" -71	-74
EPC*55	53.7	" -62	-71
" 45	55.4	" -65	-72
" 35	59.0	" -67	-72
SAF 55	46.	" -60	-65
" 45	49.6	" -65	-67
" 35	55.9	" -72	-72

* See Page 42

TABLE XLII. TR - Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions (T-3, T-5, etc.) indicated.

Black	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
MAF 55	-	89	82	76	72	57	67	63	60	55	43
" 45	-	89	81	76	73	71	68	66	62	58	47
" 35	-	88	84	80	77	74	72	69	65	60	51
EPC 55	88	84	81	76	73	71	67	63	58	53	36
" 45	-	88	85	79	74	72	68	64	62	55	41
" 35	-	87	83	79	74	72	69	65	62	56	42
SAF 55	-	84	82	74	70	65	62	58	53	45	30
" 45	87	85	81	75	71	67	63	60	54	47	27
" 35	-	87	84	80	76	72	69	65	62	54	38

I. VARIABLE ZINC OXIDE IN BUTYL

Formula

GR-I 18	100.
MAF Black	55.
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2.
Adipol 2 EH	20.
Zinc Oxide	Variable
Curing temperature	287°F.

TABLE XLIII.

Parts Zinc Oxide Per 100 GR-I	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch					
3	175	525	675	800	875
5	160	540	680	825	910
10	150	500	650	800	900
20	100	450	625	800	875
Tensile @ Break - Pounds per square inch					
3	650	1975	1825	1725	1625
5	1070	1910	1870	1750	1650
10	1150	1750	1625	1650	1650
20	900	1675	1650	1675	1550
Elongation @ Break - Per cent					
3	775	710	650	580	540
5	800	680	640	565	515
10	870	645	580	535	505
20	935	680	595	550	510
Tear - Pounds per inch					
3	103	203	200	178	160
5	150	236	200	174	169
10	133	226	218	198	167
20	107	216	199	194	192
Hardness (Shore Durometer Type A)					
3	36	44	46	49	51
5	37	44	47	49	51
10	37	45	48	50	52
20	38	44	48	51	52

TABLE XLIV.

Rebound (Goodyear-Healy) 60 Min. @ 287°F.

Embrittlement (American Cyanamid-Graves) 60 Min. @ 287°F.

TR (40% Retraction - Original Elongation 50%) 60 Min. @ 287°F.

Parts Zinc Oxide Per 100 GR-I	Rebound	Embrittlement	TR
3	57.4%	OK @ -67°C.	-63°C.
5	58.4	" -71	-69
10	57.9	" -63	-69
20	56.9	" -63	-69

TABLE XLV. TR = Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions (T-3, T-5, etc.) indicated.

Parts Zinc Oxide Per 100 GR-I	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
3	89	87	83	78	68	63	60	58	55	54	47
5	89	85	82	75	68	69	64	58	55	51	44
10	89	87	85	76	72	69	66	53	58	52	44
20	89	87	85	76	72	69	66	63	58	53	45

J. VARIABLE ISOPRENE CONTENT IN BUTYL

Formula

Butyl*	100.
Zinc Oxide	5.
MAF Black	55.
Captax/Tuads (1:2 Blend)	1.54
Sulfur	2.
Adipol 2 EH	20.
	183.54

* Grades of butyl investigated are as follows:

	% Isoprene
GR-I 35	1.0
GR-I	2.0
GR-I 15	2.5
GR-I 25	3.0

TABLE XLVI.

Butyl	Per Cent Isoprene	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch						
GR-I 35	1.0%	NC	325	425	550	650
GR-I	2.0	250	450	550	750	875
GR-I 15	2.5	250	575	750	900	1025
GR-I 25	3.0	400	700	850	1050	1150
Tensile @ Break - Pounds per square inch						
GR-I 35	1.0%	NC	1525	1600	1600	1775
GR-I	2.0	1250	1750	1750	1650	1600
GR-I 15	2.5	1450	1725	1750	1575	1575
GR-I 25	3.0	1525	1600	1500	1450	1425
Elongation @ Break - Per cent						
GR-I 35	1.0%	NC	730	675	650	615
GR-I	2.0	800	720	675	565	515
GR-I 15	2.5	820	685	635	515	460
GR-I 25	3.0	780	625	530	425	375

NC indicates no cure.

TABLE XLVI. (Contd.)

Butyl	Per Cent Isoprene	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Tear - Pounds per inch						
GR-I 35	1.0%	NC	199	236	238	216
GR-I	2.0	210	240	236	222	184
GR-I 15	2.5	218	226	220	181	163
GR-I 25	3.0	246	212	194	172	160
Hardness (Shore Durometer Type A)						
GR-I 35	1.0%	NC	39	42	44	46
GR-I	2.0	38	44	46	48	49
GR-I 15	2.5	40	46	48	51	42
GR-I 25	3.0	42	47	49	52	55

TABLE XLVII.

Rebound (Goodyear-Healy) 60 @ 287°F.
Embrittlement (American Cyanamid-Graves) 60 @ 287°F.
TR (40% Retraction - Original Elongation 50%) 60 @ 287°F.

Butyl	Per Cent Isoprene	Rebound	Embrittlement	TR
GR-I 35	1.0%	49.1%	-71°F.	-68°F.
GR-I	2.0	52.	-71	-67
GR-I 15	2.5	54.9	-71	-68
GR-I 25	3.0	59.0	-72	-70

TR at 40% Retraction - Comparison of versus State of Cure		20 Min.	30 Min.	45 Min.	60 Min.
GR-I 35	1.0%	-63°F.	-67°F.	-68°F.	-68°F.
GR-I	2.	-64	-68	-68	-67
GR-I 15	2.5	-66	-69	-69	-68
GR-I 25	3.0	-68	-71	-71	-70
Embrittlement - Comparison of versus State of Cure					
GR-I 35	1.0%	-65°F.	-67°F.	-69°F.	-71°F.
GR-I	2.	-69	-67	-69	-71
GR-I 15	2.5	-69	-67	-69	-71
GR-I 25	3.0	-68	-71	-71	-70

From this data it is noted that variation of TR and Embrittlement at different states of cure is very slight in butyl compounds. Therefore, unless extreme conditions in cure rate are encountered, comparisons based on a single cure are adequate.

NC indicates no cure.

TABLE XLVIII. TR - Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions (T-5, T-10, etc.) indicated.

Butyl	% Isoprene	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
					20 @ 287°F.						
GR-I 35	1.0	86	82	74	68	63	56	45	35	19	+2
GR-I	2.0	85	81	74	69	64	60	54	45	33	12
GR-I 15	2.5	86	82	76	71	66	63	57	51	42	23
GR-I 25	3.0	87	83	78	72	68	64	61	45	49	33
					30 @ 287°F.						
GR-I 35	1.0	87	83	78	72	67	63	56	45	33	12
GR-I	2.0	86	82	77	72	68	65	60	54	41	24
GR-I 15	2.5	87	83	78	73	69	66	62	58	49	31
GR-I 25	3.0	87	84	80	74	71	67	65	60	54	40
					45 @ 287°F.						
GR-I 35	1.0	87	83	77	72	68	64	59	53	44	27
GR-I	2.0	86	82	76	72	68	64	61	55	48	33
GR-I 15	2.5	87	83	77	72	69	66	62	58	53	41
GR-I 25	3.0	87	86	79	74	71	67	63	61	55	44
					60 @ 287°F.						
GR-I 35	1.0	87	83	76	72	68	63	59	52	44	26
GR-I	2.0	86	81	75	71	67	63	60	54	48	34
GR-I 15	2.5	86	82	75	72	68	64	61	56	51	41
GR-I 25	3.0	87	83	76	72	70	65	63	59	53	44

K. PEROXIDE CURATIVES IN BUTYL

Formula

	A	B	C
GR-I 18	100.	100.	100.
Philblack E	45.	45.	45.
Zinc Oxide	5.	5.	5.
Butyl Cellosolve Pelargonate	10.	10.	10.
Monoplex DOS	10.	10.	10.
2 Ethyl Hexyl Ether	10.	10.	10.
Captax/Tuads (1:2 Blend)	1.54	-	-
Dibenzo GMF	-	6.	6.
Red Lead	-	10.	10.
Polyac	-	1.	-
Sulfur	2.	3.	3.
	183.54	200.	199.

A. Standard curative - organic acceleration.
B. Peroxide curative and Polyac.
C. Peroxide curative without Polyac.

TABLE XLIX.

Formula	2 Min.	4 Min.	6 Min.	8 Min.	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch									
A	NC	NC	NC	NC	NC	175	250	350	450
B	75	125	400	625	650	775	850	850	925
C	NC	50	275	425	475	650	650	725	750
Tensile @ Break - Pounds per square inch									
A	NC	NC	NC	NC	NC	2150	2400	2400	2350
B	400	775	1300	1475	1325	1200	1350	1375	1325
C	NC	375	1175	1400	1500	1400	1400	1350	1400
Elongation @ Break - Per cent									
A	NC	NC	NC	NC	NC	865	855	715	705
B	885	675	510	475	445	365	370	425	385
C	NC	840	630	545	515	445	455	455	445
Tear - Pounds per inch									
A	NC	NC	NC	NC	NC	344	310	236	218
B	51	95	139	167	129	111	114	102	115
C	NC	47	116	171	141	142	117	136	110
Hardness (Shore Durometer Type A)									
A	NC	NC	NC	NC	NC	35	38	40	41
B	30	32	41	43	45	48	48	48	49
C	NC	26	35	42	44	48	48	48	48

NC indicates no cure.

TABLE L.

Rebound (Goodyear-Healy) 60 @ 287°F.
Embrittlement (American Cyanamid-Graves) 60 @ 287°F.
TR (40% Retraction - Original Elongation 50%) 60 @ 287°F.

Formula	Rebound	Embrittlement	TR
A	60.0%	OK @ -80°F.	-81°F.
B	60.0	" -81	-79
C	57.9	" -80	-78

TABLE LI.

Mooney Scorch:

MS @ 250°F.

Minutes	Formula A.	Formula B.	Formula C.
1	13	22	15
2	12	25	14
3	12	28	13
4	11	33	13
5	11	36	14
6	11	39	15
7	11	41	15
8	11	44	16
9	11	46	17
10	11	48	18
11	12	50	18
12	12		21
13	12		23
14	12		26
15	13		31
16	13		36
17	13		40
18	14		44
19	14		47
20	15		51
21	16		
22	17		
23	18		
24	20		
25	23		
26	27		
27	30		
28	33		

If the scorch time is taken as the time required to reach a viscosity of 5 points greater than the minimum, then scorch time is:

A	21 minutes
B	2½ minutes
C	10 minutes

TABLE LII. TR - Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions (T-10, T-20, etc.) indicated.

Formula	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
A	87	86	83	81	77	73	67	57	31
B	88	86	83	79	74	67	58	41	7
C	87	85	82	78	72	65	54	35	+3

Time limitations on this contract did not permit obtaining TR data after 4 weeks migration at 158°F.

L. HIGH TEMPERATURE (400°F.) PROCESSING

Formula

	A	B	C
GR-I 18	100.	100.	100.
Philblack E	40.	40.	40.
Polyac	-	0.4	0.4
Stearic Acid	-	0.4	0.4
Zinc Oxide	5.	5.	5.
Monoplex DOS	25.	25.	25.
Captax/Tuads (1:2 Blend)	1.54	1.54	1.54
Sulfur	2.	2.	2.
	173.54	174.34	174.34

Remarks:

On A and B the preparation and handling of batches was exactly as described in this report in the section "Testing Methods and Procedures." Discharge temperatures of the batches from the Banbury mixers were 220-230°F.

Formulation for C is identical to B the difference between these two being in mixing. Method of handling C follows: The initial mix consisted of a masterbatch which was processed in the Banbury at high temperature. Formula for this masterbatch is:

GR-I 18	100	or	80
Philblack E	50		40
Polyac	0.5		0.4
Stearic Acid	0.5		0.4

This batch was mixed in the laboratory Banbury for 10 minutes after batch temperature had reached 400°F. Overall mixing time for this batch was 16 minutes. Stearic Acid was added just before batch discharge in order to assist in lubrication and prevent sticking while resheeting. After two hours for cooling, the batch was remilled, again in the Banbury, but in this step at normal processing temperatures, 200-220°F. In this step, additional GR-I 18 was added in order to reduce black content from 50 parts per 100 GR-I 18, the concentration used in the hot mix, to 40 parts per 100 GR-I 18 which is the concentration in the final mix. At this stage plasticizer and zinc oxide were added. After allowing two to four hours for cooling the batch was finished by regular procedure, that is, sulfur and accelerator added on 6 x 12 mill. It should be mentioned that the above method of processing butyl rubber is on recommendations supplied by Esso Laboratories. This method of processing purported to result in improved physical properties.

TABLE LIII.

Formula	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch					
A	25	200	325	450	525
B	100	325	525	600	725
C	50	400	625	775	900
Tensile @ Break - Pounds per square inch					
A	825	2725	2775	2850	2750
B	1575	2450	2450	2625	2325
C	425	2075	2175	2275	2200
Elongation @ Break - Per cent					
A	865	875	775	730	665
B	900	750	665	630	560
C	1115	660	550	540	495
Tear - Pounds per inch					
A	141	350	354	254	258
B	187	344	318	292	195
C	41	232	248	216	202
Hardness (Shore Durometer Type A)					
A	30	36	39	41	42
B	32	37	41	42	43
C	25	34	38	40	41

Rebound (Test piece 2" x 1" x 1") cured 60 Min. @ 287°F.

	% Rebound		Shore Hardness*	
	@ Room Temperature	@ 212°F.	@ Room Temp.	@ 212°F.
A	55.4%	76.3%	42	42
B	57.9	76.9	42	40
C	59.0	77.5	40	37

* This data obtained on 2" x 1" x 1" blocks. Shore Hardness on thicker test pieces is usually a point or more lower than the data obtained on tensile sheets.

TABLE LIV.

Physical Properties at 212°F.

Formula	10 Min.	20 Min.	30 Min.	45 Min.	60 Min.
Modulus @ 300% - Pounds per square inch					
A	25	125	200	300	350
B	75	200	250	375	475
C	-	175	275	425	575
Tensile @ Break - Pounds per square inch					
A	300	975	925	575	575
B	475	975	825	700	675
C	-	750	725	675	700
Elongation @ Break - Per Cent					
A	900	950	770	470	400
B	1000	795	585	415	350
C	-	615	475	380	325
Tear - Pounds per inch					
A	107	134	138	126	135
B	129	155	150	117	130
C	-	120	113	133	101

TABLE LV.

Rebound (Goodyear-Healy) 60 @ 287°F.

Embrittlement (American Cyanamid-Graves) 60 @ 287°F.

TR (10% Retraction - Original Elongation 50%) 60 @ 287°F.

Formula	Rebound	Embrittlement	TR
A	55.4%	OK @ -71°F.	-73°F.
B	57.9	" -71	-75
C	59.0	" -76	-76

TABLE LVI. TR - Temperature Retraction Data

Temperature data in minus degrees Fahrenheit for percentage retractions (T-1, T-2, etc.) indicated.

Formula	T-1	T-2	T-3	T-5	T-10	T-20	T-30	T-40	T-50	T-60	T-70	T-80	T-90
A	-	-	-	88	86	82	78	73	69	65	61	54	33
B	-	-	-	88	86	83	80	75	71	67	62	55	40
C	-	-	-	88	87	83	81	76	72	67	63	56	41

TR - Temperature Retraction after 4 weeks migration @ 158°F.

A	86	85	81	78	71	62	56	54	47	43	36	27	0
B	-	88	86	82	74	66	61	57	54	48	43	35	18
C	-	86	85	81	74	66	61	57	55	49	44	36	22

M. VOLATILITY OF PLASTICIZERS

TABLE LVII.

Data indicates per cent of plasticizer lost or volatilized under conditions indicated.

	Plasticizer	Compounded Stock							
Plasticizer	5 Hours @ 325°F.	70 Hours @ 212°F.				48 Hours @ 300°F.			
Parts Plasticizer Per 100 GR-I 18	-	15	20	25	30	15	20	25	30
Forum 40 Oil	45.9%	46.1%	39.0%	42.7%	-	83.2%	84.1%	87.1%	-
C. P. Hall 3890-A	7.2	-	10.5	10.8	10.4	-	76.8	95.4	86.7
Adipol 2 EH	22.2	-	53.6	45.3	34.5	-	97.7	93.7	97.0
10-A Plasticizer	17.1	-	39.4	39.9	27.0	-	95.7	93.3	98.8
Di 2 Ethyl Hexyl Ether	99.1	-	46.4	44.5	-	-	77.2	68.6	-
Trioctyl Phosphate	39.1	-	48.5	29.7	-	-	71.8	75.1	-
Butyl Cellosolve Pelargonate	96.2	-	41.2	39.6	-	-	88.5	88.0	-
Butyl Carbitol Pelargonate	96.4	-	68.6	45.5	-	-	85.5	70.2	-
Diisobutyl Azelate	98.8	-	65.5	78.1	-	-	83.4	90.0	-
Monoplex DOS	5.5		0.0	0.0		-	43.2	46.4	
Hexyl Ether	100.		21.1	25.1			24.1	30.7	

Compounded stock for volatility tests cured 60 @ 287°F.

N. MIGRATION OF PLASTICIZERS

TABLE LVIII

Migration temperature 158°F.
Area of test piece 2" x 1"
Migration pressure 1 kilogram/sq. in.
Cure of stock 60 @ 287°F.
Data shows % plasticizer lost through migration after number of days indicated.

Plasticizer	Parts PER 100 GR-I	Days Migration 1	2	3	4	7	14	21	28
Forum 40 Oil	15	14.6%	22.8%	27.6%	32.2%	41.9%	51.0%	56.0%	57.9%
" " "	20	12.6	17.2	21.2	25.1	31.7	44.6	48.1	49.9
" " "	25	18.6	25.7	29.8	33.9	43.9	57.1	61.8	64.0
C. P. Hall 3890-A	20	9.4	27.1	32.8	26.0	48.7	60.5	62.2	62.4
" " " "	25	15.1	27.9	31.8	36.1	51.2	62.7	68.7	70.0
" " " "	30	7.1	10.8	16.2	18.8	24.8	44.0	48.3	53.4
Adipol 2 EH	20	14.8	25.8	35.0	45.5	59.3	71.3	78.0	80.0
" "	25	23.9	32.8	41.8	47.8	58.5	71.1	74.6	75.5
" "	30	17.2	35.6	45.8	52.1	63.0	72.4	74.0	74.4
10-A Plasticizer	20	23.8	33.8	42.0	47.4	58.9	70.0	73.8	74.5
" "	25	17.8	29.1	36.0	40.5	53.7	63.7	68.4	69.0
" "	30	22.0	30.1	33.9	40.2	49.7	57.2	60.7	62.0
Di 2 Ethyl Hexyl Ether	20	9.5	12.7	14.6	15.1	14.8	14.4	12.6	11.4
" " " " "	25	8.5	11.7	13.2	14.1	14.1	13.3	11.7	10.6
Trioctyl Phosphate	20	11.7	22.7	27.3	32.1	42.0	55.0	60.2	61.4
" "	25	15.8	28.1	32.5	36.7	47.6	58.5	63.0	64.0
Butyl Cellosolve Pelarg.	20	8.5	10.3	11.0	11.3	10.5	8.7	6.8	5.5
" " "	25	8.5	11.4	12.3	12.9	12.8	12.3	13.3	9.9
Butyl Carbitol Pelarg.	20	10.1	13.5	14.6	15.1	15.2	17.6	13.6	12.6
" " "	25	11.1	15.1	16.6	17.4	18.3	18.4	18.0	17.4
Diisobutyl Azelate	20	19.2	27.3	31.5	33.8	36.8	37.1	36.0	34.8
" "	25	22.2	30.6	34.0	36.0	37.8	37.5	36.5	35.4
Monoplex DOS	20	16.7	23.0	25.2	27.5	41.3	54.6	62.5	66.5
" "	25	16.7	20.9	23.5	27.5	41.8	68.5	73.0	74.6
Hexyl Ether	20	18.1	20.1	20.5	20.5	19.5	17.7	16.8	16.3
" "	25	18.0	20.0	20.6	20.6	18.9	18.1	17.5	17.3

O. MIGRATION, EFFECT OF ON LOW TEMPERATURE PROPERTIES

TABLE LVIX.

Plasticizer	Migration Loss % of Plasticizer	Parts Per 100 RHC Before	Parts Per 100 RHC After	Embrittlement OK @ °F. Before	Embrittlement OK @ °F. After	TR °F. Before	TR °F. After
Forum 40 Oil	36.7	15	9.5	54	56	57	55
" " "	41.0	20	11.8	62	53	58	56
" " "	44.4	25	14.0	62	56	61	56
C. P. Hall 3890-A	56.8	20	8.6	54	53	67	53
" " "	61.6	25	9.6	72	53	70	53
Adipol 2 EH	56.2	20	8.8	72	53	71	58
" "	56.6	25	10.9	72	53	72	58
10-A Plasticizer	51.8	20	9.6	72	56	74	58
" "	57.4	25	10.7	74	56	72	58
Di 2 Ethyl Hexyl Ether	30.0	20	14.0	78	56	76	56
" " " " "	27.4	25	18.2	76	56	74	53
Trioctyl Phosphate	67.0	20	6.6	63	56	70	54
" "	69.2	25	7.7	76	56	72	56
Butyl Cellosolve Pelargonate	39.0	20	12.2	72	56	74	56
" " "	44.4	25	14	78	53	77	56
Butyl Carbitol Pelargonate	50.7	20	9.9	72	56	67	58
" " "	52.8	25	11.8	76	56	67	60
Diisobutyl Azelate	56.4	20	8.7	72	56	66	62
" "	56.8	25	10.8	72	56	68	60
Monoplex DOS	56.0	20	8.8	67	62	70	60
" "	83.2	25	4.5	72	62	73	60
Hexyl Ether	8.9	20	18.2	71	53	72	54
" "	13.6	25	21.6	74	56	72	56
No plasticizer	0	0	0	45	54	45	54

Note: Migration tests on 6" x 6" tensile sheets. It will be noted that migration loss on 6 x 6 sheets fails to agree with the data obtained on 2" x 1" pieces, data on which is shown in Section N.